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# Investigation of the Compressive Strength of Glass Ionomer Cement Containing Nanoparticles

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## Abstract

**Purpose**: This study evaluated the effect of $Al_2O_3$ and $SiO_2$ nanoparticles on compressive strength of glass ionomer cement.

**Materials and Methods:** Five groups of 50 disc shaped specimens were used; **Group A**:Glass ionomer cement without nanoparticle, **Group B**:Glass ionomer cement containing 3% $Al_2O_3$ nanoparticle, **Group C**: Glass ionomer cement containing 5% $Al_2O_3$ nanoparticle, **Group D**: Glass ionomer cement containing 3% $SiO_2$ nanoparticle, **Group E**:Glass ionomer cement containing 5% $SiO_2$ nanoparticle. Compressive strength tests were carried in an universal test machine with 1 mm/min. cross head speed.

**Results:** The statistical analysis was performed by one way ANOVA test and post-hoc Tukey HSD tests.GI-containing 3 % and 5% (w/w) $Al_2O_3$ nanoparticles showed significantly higher compressive strengths compared to 3 % and 5% (w/w) $SiO_2$ nanoparticles and control group.

**Conclusion:** GI-containing 3% and 5% (w/w) $Al_2O_3$ nanoparticles is a promising restorative material with improved mechanical properties. This novel experimental GI may be potentially used for higher stress-bearing site restorations.

**Keywords:** Nanoparticle, glass ionomer cement, compressive strength.

## Introduction

Glass ionomer cement (GIC) for dental restorative applications are formed by an acid-base reaction between calcium fluoro-alumino- silicate glass and polyacrylic acid. Since their introduction in 1972 to the dental field they have been widely used as restorative materials, sealant, luting cement and cavity base materials. Glass ionomers have certain exceptional properties such as chemical adhesion to dental hard tissues, anticariogenic/antibacterial properties from the release of fluoride, good thermal compatibility with tooth structure and acceptable biocompatibility. However GICs have some disadvantages or limitations including early moisture sensitivity, brittleness and inferior mechanical strength when compared to resin- based restorative materials [1].

One of the major drawback is low fracture strength and increased occlusal wear rate when compared with other restorative materials. Thus special attention is needed to be looked into the mechanical properties of the GIC. Over the period they have undergone constant improvement in order to fulfil the current market trends and also to satisfy the function and aesthetic representation. They are also inexpensive when compared to resin composite restorations. Thus mechanical properties of GIC have been investigated since their development and concerns have remained even after the development of resin modified GIC (RMGIC) [2].

Glass ionomer cements were introduced as hybrids of silicate cements and polycarboxylate cements to have characteristics of fluoride release (from silicate cements) and adhere to enamel and to some extent to dentin (from polycarboxylate cements). It is noteworthy that the physical properties of conventional glass ionomer cement can be highly variable based upon different powder/liquid ratios so mixing should be adhered to according to the manufacturers' instructions [3].

GIs although often used as restorative materials cannot generally withstand the forces generated in the posterior area of the mouth because of their low mechanical properties, especially the low fracture toughness [4-6]. Usually the performance standards for assessing restorative materials usually involve the measurements of compressive strength, diameter tensile strength, flexural strength, flexural modulus, color stability, fluoride release and adhesive bond strength [7].

Compressive tests are used in dentistry for laboratory simulation of the stress that may result from forces applied clinically to a restorative, base/liner or core build material. Most mastication forces are compressive in nature but exact critical value is unknown [8].

The hybrid system of nanoparticles dispersed in polymer matrix has received extensive attention recently [9,10]. As a result, nanoparticle-reinforced hybrid system exhibits higher stiffness and better resistance to wear [11]. In restorative dentistry, there has also been a growing interest in using nanoparticles to improve properties of dental restoratives [12]. Particles sized between 1-100 nm are qualified as nanoparticles and possess a large active surface, which enables them to transfer loads acting on the polymer's surface. Thanks to this, the material gains stiffness and physical resistance [13]. However, little work has been reported so far regarding use of any nanoparticles to improve dental glass-ionomer cement (GIC) [8,14]. Although GIC has demonstrated many advantages over dental composite resins, its wear resistance and most mechanical strengths are lower than dental composite resins [15]. Part of the reason has been attributed to the nature of the weak sintered glass fillers in GIC [16,15]. Several attempts have been made to improve the performance of GIC fillers including adding filler particles such as hydroxyapatite [17], zirconia (ZrO2) [18], YbF3/BaSO4 [19], silver [20], etc into GIC. However, none of them have demonstrated significantly improved wear resistance and mechanical strengths. Nanoparticles of silica, alumina, and titania have been successfully used in biomedical materials research for improved wear resistance and mechanical strengths [21-23]. The large specific surface areas of these nanoparticles were believed to facilitate the transfer of loading force from polymer matrix to nanoparticles, thus leading to better resistance to wear [24,11]. It was found that degree of separation and uniformity of distribution of the nanoparticles was the key to mechanical strengths [23]. Therefore, in this study we hypothesize that these nanoparticles might improve wear resistance and surface hardness of dental GIC [25].

Nanotechnology also provides some value-added features not typically associated with glass ionomer restorative materials such as improved polish and aesthetics, abrasion resistance, strength, optical properties and increased fluoride release [26].

In this work we aim to come over the disadvantage of GIC by incorporating nano-silica and $Al_2O_3$ with different quantities. The objective of this researchwas to investigate the compressive strength of glass ionomer cement containing 3% and 5% $Al_2O_3$ and $SiO_2$ nanoparticles.

The null hypothesis of this study was thatthere were no differences in the compressive strengths following the addition of nanoparticles.

## Material and Methods

### Specimen preparation

In this study conventional glass ionomer cement (MERON, Germany) were used. 3%, 5% $SiO_2$ and $Al_2O_3$ nanoparticles were added to glass ionomer powder as shown in Fig 1. The specimen of each group was prepared as shown in Fig.1. Used materials and manufacturers were given in Table 1. Specimens were divided into five groups. The specimen groups were shown in Table 2. Every group contained 10 specimens (n=10). Total of fifty specimens were prepared in dimensions of 4 mm diameter and 6 mm height.

### Compressive strength test

Compressive tests were performed in an universal test machine with 1 mm/min. cross head speed. Compressive loading were applied until specimen is get to be broken and compressive load values were recorded. Compressive strength values were calculated by Equation 1. Where σ (MPa) is compressive strength, F (N) is compressive load at fracture and d (mm) is specimen diameter.

$$\sigma = \frac{4F}{\pi d^2} \qquad \text{Eq. 1.}$$

### Statistical analysis

The statistical analysis was performed using a software package (SPSS 10.0 for Windows, SPSS, Inc., Chicago, USA). Means and standard deviations for the chosen parameters were calculated. The analysis comprised ofone way analysis of variance and post-hoc Tukey HSD tests.

## Results

Mean and standard deviations of compressive strength for groups were given in Fig.2. Data showed a significant increase in compressive strength for the GI cement containing 5% (w/w) $Al_2O_3$ NPs compared to the control group and 3% and 5% (w/w) $SiO_2$group.There was significant difference between GI cement containing 3% (w/w) $Al_2O_3$ control group and 3% $SiO_2$ group. There was no significant difference between control and 3% and 5% (w/w) $SiO_2$ group. The ratio guantity (3% and 5%) had no significant effect on compressive strength for both $Al_2O_3$ and $SiO_2$ groups.The compressive strength values of 3% $SiO_2$ were significantly smaller than 3% and 5% (w/w) $Al_2O_3$ group. On the other hand there was no significant difference between 3% $Al_2O_3$ and 5% $SiO_2$ group.

## Discussion

In recent decades, glass ionomer cements (GICs) have been widely used as a luting agent, a cavity liner and for bases and restorative material. Some important characteristics of GICs include fluoride ion release, a low thermal coefficient of expansion almost near the tooth, the bonding ability to the tooth and metallic surfaces and biocompatibility [27]. However, its use is associated with problems like high technique sensitivity and lower esthetics compared to composite resins. Furthermore due to its low compressive strength and mechanical properties it can not be applied as a restorative material at areas under occlusal stres [28]. Low resistance to fracture of glass-ionomers is commonly attributed to the presence of porosity in the cement matrix. These are formed by the inclusion of air during mixing [29]. Once the material sets, these voids become trapped in the cement where they act as stress concentrations and thus points of mechanical weakness [30] between both of the tested materials, or between the different types of nanoparticles. Elizabeta Gjorgievska et al. [31] found that although the nanoparticles were of different sizes, there were no noticeable differences in their behavior and all appeared to blend readily into the cement and to reduce the porosity when set as we concluded in our study.

In Elizabeta Gjorgievska et al's study [31],compressive strength results (Table 3) indicate that the nanoparticles had no adverse effect on mechanical integrity of the cements as we found. Materials without added nanoparticles hadsubstantial porosities and microcracks within them and had compressive strengths as 32 MPa which we found as 21,6 MPa.

The present study involves two different types of nanoparticles, namely $Al_2O_3$ and $SiO_2$. They were selected because they exhibit relatively low toxicity when present in other dental restorative formulations [32,33,34]. In addition, compressive strength investigation werecarriedouton 3 and 5 wt % additions of the different nanoparticles; hereby presented are the results of the samples with 5wt% addition of $Al_2O_3$ nanoparticle, which showed the best performance (Table 3). $SiO_2$, $Al_2O_3$, or $TiO_2$ particles are tougher than the sintered filler particles in GIC. Therefore, under an indentation loading, these particles would undergo elastic rather than plastic deformation, as compared to GIC [25].

Elizabeta Gjorgievska et al [31] also used specimens containing $Al_2O_3$, $ZrO_2$, and $TiO_2$ nanoparticles have substantially different microstructure than the original cements. Occurrence of the air voids is diminished, and those that do occur appear shallower than those in the unmodified cement. There are fewer cracks in the matrices ofthe modified cements and their lengths and widths are reduced compared with the cracks in the unmodified cementstypical for conventional GICs, namely aluminum, fluorine, zinc, phosphorus, silicon, strontium, magnesium, and calcium which can be correlated with the higher compressive strength for $Al_2O_3$ as we found [31].

Khademolhosseini et al., 2012 [35] used the nanoparticles' ratio as we preferred in our study from 1 to 3 wt.% has led to a moderate increment of compressive strength, the specimens containing 5 wt.% $TiO_2$ have demonstrated more highlighted enhancement in the value of compressive strength as well as a higher diametrical tensile strength and microhardness values compared to the others [35]. In the light of this study, future investigations can be done with $TiO_2$ nanoparticle.

The compressive and diametral tensile strengths of $Al_2O_3/TiO_2$–GICs was, however, significantly greater than another GICs due to the better particle size distribution and interfacial bonding between the particles and the matrix inKhademolhosseini et al's study [35 ]as it was in our study.

The mechanical strength is found to be dependent upon the particle size and particle size distribution of restorative materials [36]. As a result, the composition of glass particles with a larger particle size and $Al_2O_3/TiO_2$ particles with nano size particles has led to a wide distribution of the particle size. This would allow a high packing density of the mixed particles within the glass ionomer matrix [36]. On the other hand, the wide distribution of nano sized particles within the larger glass particles can generate a high packing density of glass ionomer cement with an enhanced mechanical strength as we similarly found higher compress've strengths for 5% $Al_2O_3$ nanoparticles. However, lower amount of voids as well as good distribution of nano powders in Al2O3/TiO2-GIC can result in the highest amount of mechanical properties [35].

In addition to particle size and particle size distribution, the cross-linking formation during setting plays an important role on the final mechanical properties of the cements [36,37]. An acid-base reaction occurs during the setting procedure and forms a salt hydrogel, acting as the binding element in matrix within which the glass plays a reinforcing role. Releasing metal ions can take place upon the introduction of acid into the powders and the released metal ions operate as cross-linking species, allowing the formation of stable cement [35].

Gjorgievska et al. 2015 [31] also added 10wt% aluminum oxide, zirconium oxide and titanium dioxide nanoparticles to two commercial glass ionomer cements (ChemFil® Rock and EQUIA™ Fil). Compressive strengths of control group (without nanoparticles), group with 10% $Al_2O_3$ nanoparticles, were 33.0, 33.3 MPa respectively for ChemFil® Rock. Compressive strengths of control group (without nanoparticles) and group with 10% $Al_2O_3$nanoparticles were 32.3 and 34.0MPa respectivelyfor EQUIA™ Fil. We added 3wt% and 5wt% aluminum oxide to a glass ıonomer cement Compressive strengths of control group (without nanoparticles), group with 3% and 5% $Al_2O_3$ nanoparticles, were 21.05,42.4, 47.7 MPa respectively for (MERON, Germany).

Gu et al. [37] also state that better interfacial bonding between the particles and the matrix; propose that the aluminum and zirconium ions may have reacted with the Polyacrylic acid, forming the cross-linking in the YSZ-GIC samples. Similarly, it seems that good interfacial bonding as well as crosslinking between $Al_2O_3$nano particles and the cement matrix, resulted from the formation of aluminum salt bridges among trivalent aluminum ions in the glass as well as aluminum and titanium ions from the nano powders, provides the final strength of $Al_2O_3$ -GICs. Thereby, this may present another reason for the increase of mechanical properties in GICs as a result of applying $Al_2O_3$ nano particles in our study. However, ionic interactions and ionic bonding between the reinforcing particles ($Al_2O_3$ and/or TiO2) as well as the polyacrylic acid seem to be in need of additional investigations [35].

**Conclusion**

In general the addition of 3% and 5% $Al_2O_3$ enhanced the mechanical properties of the GIC,

According to test results the following results were found;

1. GI-containing 5% (w/w) $Al_2O_3$ nanoparticles is a promising restorative material with improved mechanical properties. This novel experimental GI may be potentially used for higher stress-bearing site restorations.
2. The ratio (3 % -5% ) amount had no difference on the compressive strength of GI cement groups.
3. The addition of 3% and 5% $SiO_2$ had no effect on the compressive strength of GI cement.

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Table 1: Properties of glass ionomer cement and nanoparticles

| Material | Manufacturer |
|---|---|
| Glass ionomer luting cement | MERON, Germany |
| $Al_2O_3$ nanopowder (99.5% pure, powder size 40-50 nm) | MKNANO, Canada |
| $SiO_2$ nanopowder coated with silane coupling agent (99.5% pure, powder size 15 nm) | MKNANO, Canada |

Table 2: Specimen groups

| Groups | Description |
|---|---|
| A | Glass ionomer cement without nanoparticles (control group) |
| B | Glass ionomer cement containing %3 wt $Al_2O_3$ nanoparticles |
| C | Glass ionomer cement containing %5 wt $Al_2O_3$ nanoparticles |
| D | Glass ionomer cement containing %3 wt $SiO_2$ nanoparticles |
| E | Glass ionomer cement containing %5 wt $SiO_2$ nanoparticles |

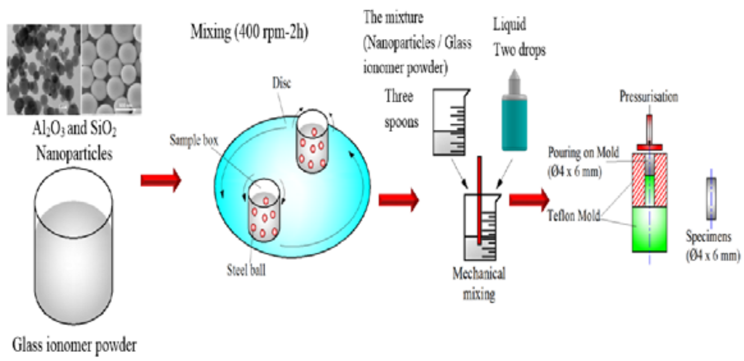


Figure 1. The preparation procedure of samples

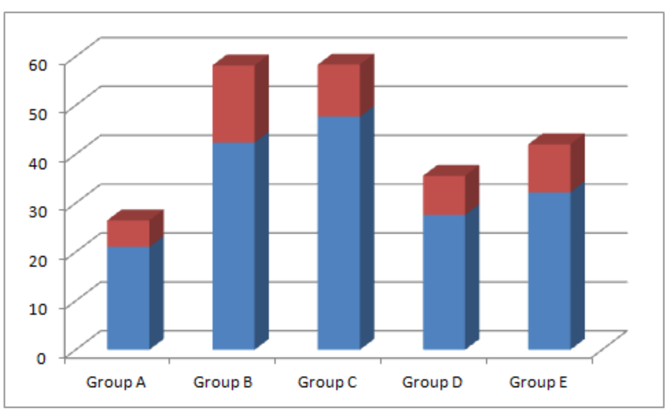


Figure 2. The mean and standard deviations of groups